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Processing and Properties of 18Ni Maraging Steel by Powder Metallurgy

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ERNEST P. ABRAHAMSON, II
MATERIALS APPLICATION DIVISION

February 1973

ARMY MATERIALS AND MECHANICS RESEARCH CENTER
Watertown, Massachusetts 02172

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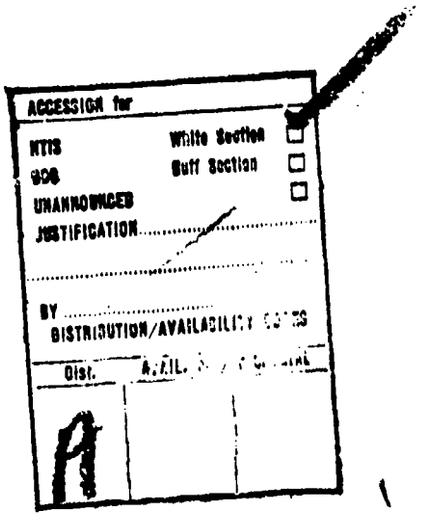

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13. ABSTRACT

Using 18Ni Mar 300 powder processed by the rotating electrode process, rods have been extruded under varying temperatures and ram speeds. It is observed that control of both variables allows structure control and increases the Charpy V-notch toughness from 7 to 23 ft-lb at room temperature. (Author)

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PROCESSING AND PROPERTIES OF 18Ni MARAGING STEEL BY POWDER METALLURGY

Technical Report by
ERNEST P. ABRAHAMSON, II

February 1973

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CONTENTS

INTRODUCTION

The alloy compositions of the commercial 18% Ni-Mo-Co magaging steels were designed to provide maximum toughness at strength levels of 200 to 350 ksi. They owe their unique properties to a tough ductile iron-nickel martensite structure which age hardens at approximately 900 F.[1] The primary precipitate responsible for the strengthening is Ni_3Mo.[2-4] Cobalt enhances the effect of Ni_3Mo by decreasing the solubility of molybdenum in the matrix.[5,6] Ni_3Ti has been identified in maraging alloys, but is believed to be a secondary precipitation hardening reaction.[4,7,8]

Strength in these materials is increased by raising the titanium content in base compositions that are progressively slightly enriched in nickel, cobalt, and molybdenum to maintain the optimum toughness. At and above 300 ksi yield strength the enriched compositions are subject to austenite retention at regions of alloy segregation.

This report deals with using rotating-electrode-processed powder in an effort to reduce segregation and improve properties.

PROCEDURE

Commercial 300 18% nickel maraging steel was converted to spherical powders using the rotating electrode method. The composition of the alloy was as follows: 0.012 C, 0.05 Si, 0.07 Mn, 0.006 S, 0.004 P, 4.80 Mo, 8.80 Co, 18.05 Ni, 0.05 Ca, 0.10 Al, 0.70 Ti, 0.003 B, 0.009 Zr. The powders were divided into four equal weights and blended in a V-cone blender prior to being placed in the extrusion can, evacuated, and sealed.

All billets were heated at 1500 F for 3 hours and compacted under 900 tons pressure, ejected, quenched, and the extrusion can remachined.

The four billets were extruded with a ten-to-one reduction followed by a water quench. The temperatures and other extrusion data are shown in Table I.

Table I. EXTRUSION DATA

Billet	Extrusion Temperature, deg F	Ram Speed, in./min	Extrusion Force, Tons		Extrusion Constant psi	
			Upset	Running	Upset	Running
1	1650	100	825	735	35.7	32.6
2	1400	100	1110	950	47.7	40.8
3	1400	220	1110	965	47.7	41.5
4	1400	400	1130	1000	48.5	43.0

The specimens were tested in three conditions:

Treatment A - as extruded and aged three hours at 900 F

Treatment B - as extruded, solutionized at 1500 F for one hour, water quenched and aged three hours at 900 F

Treatment C - as extruded, solutionized at 1700 F for one hour, water quenched, solutionized at 1500 F for one hour, water quenched and aged three hours at 900 F.

The latter two treatments are those used on conventional wrought 18Ni Mar 300 maraging steel.

All materials were investigated metallographically using both light and transmission electron microscopy. The etch used for light microscopy was 15 cc HCl, 10 cc acetic acid, 10 cc HNO_3, and two drops of glycerin. The specimens for transmission microscopy were thinned using the window method at -40 F in a solution of 30 cc perchloric acid, 175 cc 1-butanol, and 275 cc methanol at 6 volts and 0.05 amperes.

MICROSTRUCTURE

The structure of the rotating-electrode-processed powder is shown in Figure 1. The dendrite arm spacing is approximately 3 microns. Throughout the particle one can see blocky precipitates of TiC and TiN[9] randomly dispersed.

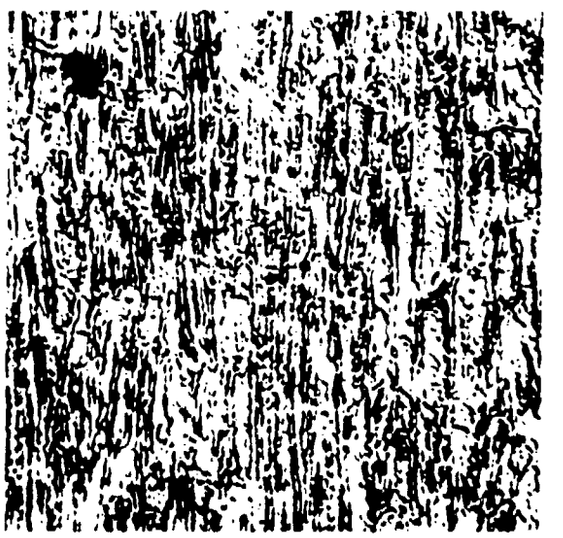
When consolidated and extruded the powder structure is completely lost, see Figure 2. The 1600 F extrusion is recrystallized and the blocky precipitates are still randomly dispersed. However, at 1450 F and 100 in./min the grains are elongated and the precipitate appears in stringers. Additionally, a second precipitate, Ni_3Mo, has started to appear. As the speed of extrusion was increased to 400 in./min at 1450 F the structure becomes a combination of elongated and recrystallized grains, with no indication of Ni_3Mo and a decreasing number of TiN and TiC stringers. This latter observation is emphasized by aging the extruded structure for three hours at 900 F, see Figure 3. In the lower temperature extrusions there is more massive precipitation of Ni_3Mo along the bands of TiN than in the 1600 F extrusion.

Figure 1. Microstructure of 18Ni Mar 300 Rotating-Electrode-Processed Powder. 1000X

a. 1600 F - 100 in./min

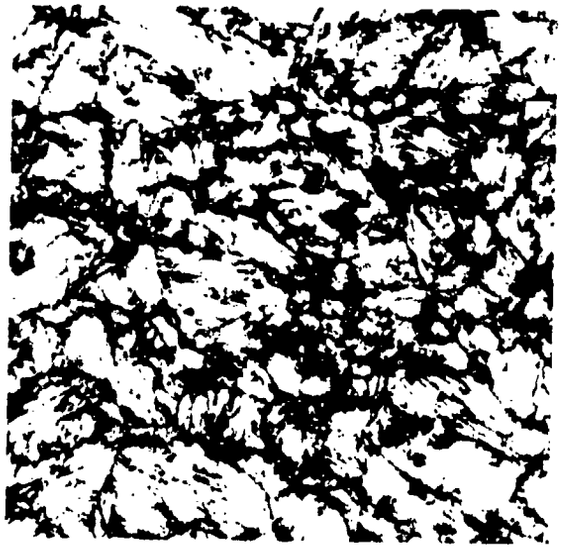
b. 1450 F - 100 in./min

c. 1450 F - 220 in./min

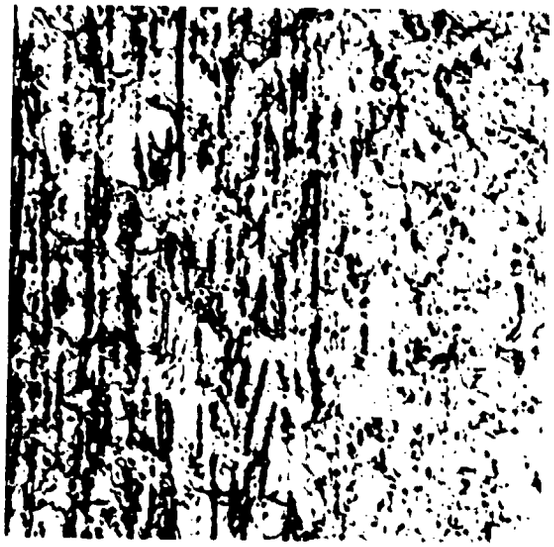


d. 1450 F - 400 in./min

Figure 2. Photomicrographs of As-Extruded 18Ni Mar 300 Steel. Mag. 1000X



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min

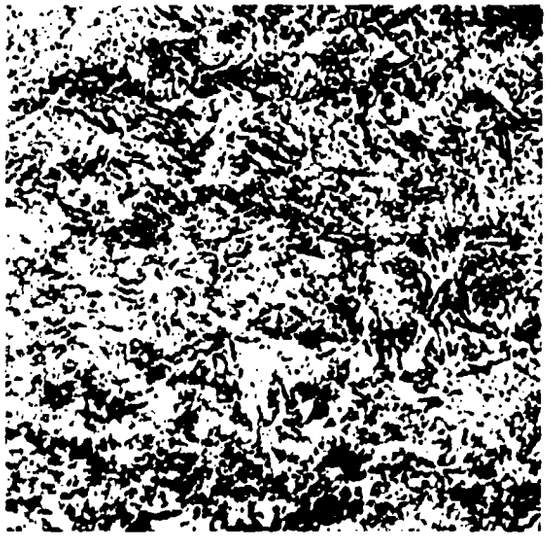
c. 1450 F - 220 in./min

d. 1450 F - 400 in./min

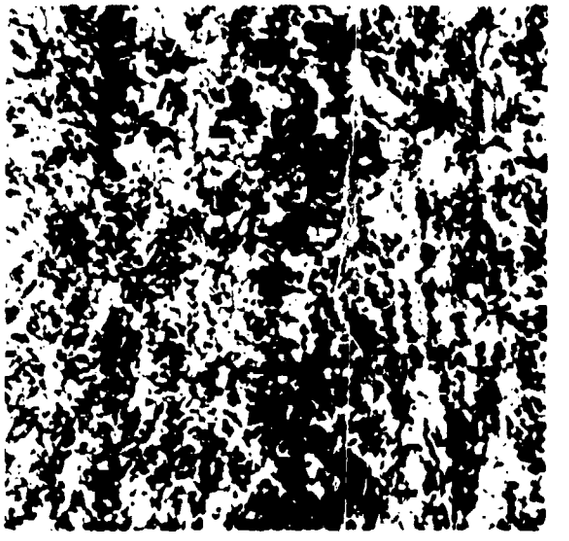
Figure 3. Photomicrographs of 18Ni Mar Steel, As-Extruded and Aged at 900 F for 3 Hr. Mag. 1000X

Three extrusions were selected for solutionizing treatments, deleting that at 1450 F - 220 in./min, which was considered redundant. The structure of the specimens given Treatment B is shown in Figure 4. The prior austenite grain structure of all the extrusions is not as obvious as those specimens that were just aged. The martensite lath structure is considerably refined by the 1500 F solution treatment, see Figure 5. The stringers are still observable in the two 1450 F extrusions, but not quite as obvious in the material extruded at 400 in./min.

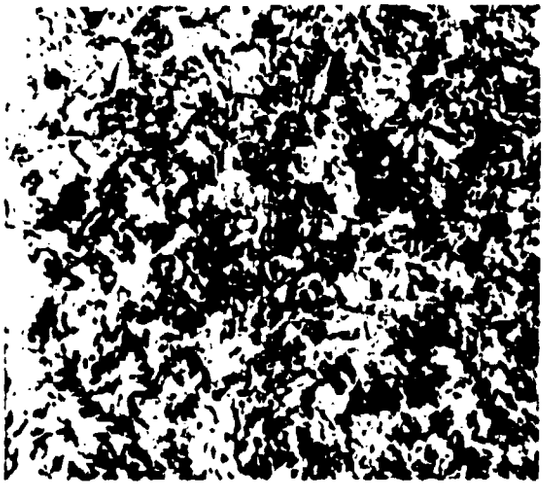
Using the higher solutionizing treatment, Treatment C, coarsens the prior austenitic and lath structure, see Figure 6. The indications of fibering are almost obliterated.



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min



c. 1450 F - 400 in./min

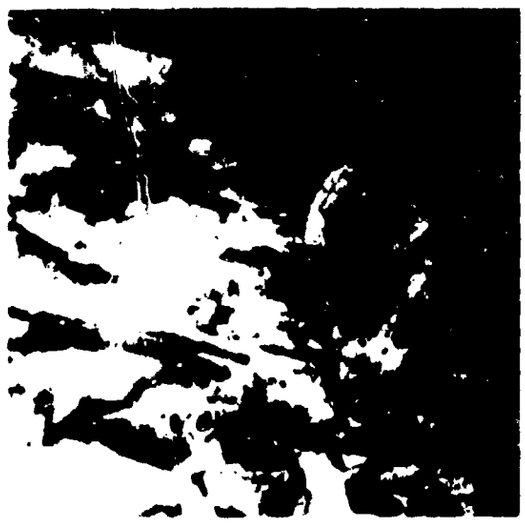
Figure 4. Photomicrographs of 18Ni Mar 300 Steel, As-Extruded, Solutionized at 1500 F for 1 Hr, Water Quenched, Aged at 900 F for 3 Hr. Mag. 500X

MECHANICAL PROPERTIES

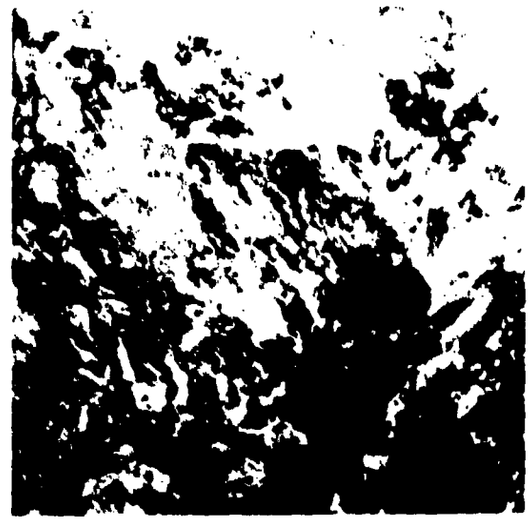
Room temperature tension tests, Charpy V-notch and hardness measurements were run on extrusions 1, 2, and 4. Extrusion 3 was omitted for the same reason noted in the previous section. The as-extruded hardness for all three was R_C 32. The properties for the heat-treated materials and a comparison with commercial material are given in Table II. Within experimental error, the hardnesses are essentially the same for all conditions.

The yield strengths in the aged and 1500 F solution-treated-and-aged conditions are slightly higher than the commercial material (at best 10%). However, the toughness of all three extrusions in these conditions equaled or bettered that of the commercial material. The as-extruded-and-aged material for the 1450 F extrusions nearly doubled the results for commercial 18Ni Mar 300, while the 1600 F extrusion more than tripled the Charpy V-notch value. That material which had an additional 1700 F solution treatment showed the lowest yield and tensile strengths, but showed an improvement in impact values over the 1500 F solution treatment.

The fracture surfaces varied with the extrusion conditions and heat treatment. The material extruded at 1600 F showed a normal fine grain fracture on the Charpy specimens. The lower temperature extrusions had fibrous fractures, with the material extruded at 400 in./min showing a combination of fibrous and fine grain fracture. With an increase in the solution treatment temperature, the fibrosity was seen to decrease.

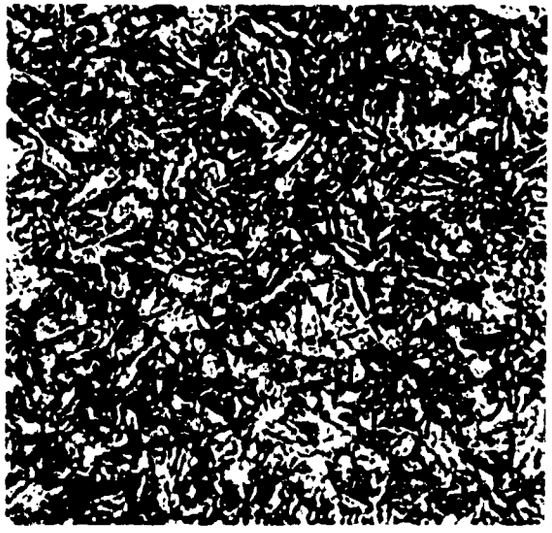


a. As Extruded and Aged at 900 F for 3 Hr

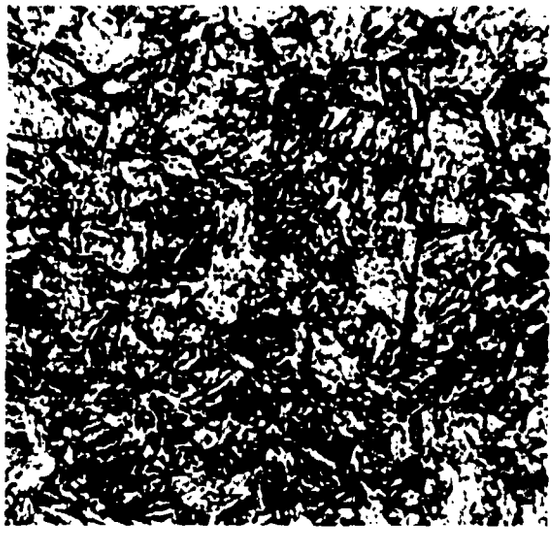


b. As-Extruded at 1500 F for 1 Hr, Water Quenched and Aged at 900 F for 3 Hr

Figure 5. Transmission Electron Micrograph of 18Ni Mar 300 Steel Extruded at 1600 F - 100 in./min. Mag. 9000X



a. 1600 F - 100 in./min



b. 1450 F - 100 in./min

c. 1450 F - 400 in./min

Figure 6. Photomicrographs of 18Ni Mar 300 Steel, As-Extruded, Solutionized 1700 F for 1 Hr, Water Quenched 1500 F for 1 Hr, Water Quenched and Aged 900 F for 3 Hr. Mag. 500X

DISCUSSION

The processing of the powders by the rotating electrode process has alleviated the problem of alloy segregation which causes "banding" in commercial material. However, the microstructures and properties indicate that care must be taken to control fibering of TiN and TiC particles. Consider the material extruded at 1600 F - 100 in./min, 1450 F - 400 in./min, and 1450 F - 100 in./min as three stages of the fibering sequence.* This indicates that the matrix must be reasonably soft, i.e., at the recrystallization temperature, to avoid fibering.

The best combination of strength and toughness was observed in all cases for the as-extruded and aged condition. Solutionizing at 1500 F plus aging refines the lath martensite structure and has a finer Ni_3Mo precipitate which further increases the strength, but decreases the toughness. Coarsening the structure by higher solutionizing temperatures reduces the strength and increases the toughness. Increasing temperature also decreases the fibering.

Table II. MECHANICAL PROPERTIES FOR 18Ni Mar 300 UNDER VARIOUS CONDITIONS

Material	Heat Treatment	0.1% Yield Stress ksi	0.2% Yield Stress ksi	Ultimate Tensile Stress ksi	True Fracture Stress ksi	Elon %	R. A. %	Charpy V-Notch Room Temp ft-lb	Hardness R_C
Commercial 300 Grade	1500 F-1hr, W.Q., 900 F-3hr, A.C.	265	266	273	381	11.5	40	7	51
REP* 300 Grade Extruded at 1650 F, 100 in./min and Water Quenched	900 F-3hr, A.C.	270	278	282	400	10	54	23	52
	1500 F-1hr, 900 F-3-hr, A.C.	282	289	293	357	10	40	11	54
	1700 F-1hr, W.Q., 1500 F-hr, W.Q., 900 F-3hr, A.C.	250	260	270	357	13	45	17	52
REP* 300 Grade Extruded at 400 F, 100 in./min and Water Quenched	900 F-3hr, A.C.	265	272	277	386	12	46	13	52
	1500 F-1hr, 900 F-3hr, A.C.	275	282	284	378	10	46	7	53
	1700 F-1hr, W.Q., 1500 F-1hr, W.Q., 900 F-3hr, A.C.	254	264	272	373	13	50	10	52
REP* 300 Grade Extruded at 1400 F 400 in./min and Water Quenched	900 F-3hr, A.C.	266	274	276	367	12	49	13	52
	1500 F-1hr, 900 F-3hr, A.C.	272	279	282	365	11	44	9	53
	1700 F-1hr, W.Q., 1500 F-1hr, W.Q., 900 F-3hr, A.C.	254	262	272	359	14	50	10	51

*Rotating Electrode Processed Powder
W.Q. - Water quenched
A.C. - Air cooled

*It is felt that 1450 F - 400 in./min falls midway between 1450 F and 1600 F due to the additional adiabatic heating at the faster rate of extrusion.

CONCLUSIONS

1. Use of rapidly cooled 18Ni Mar 300 powders alleviates alloy segregation banding.

2. Extrusion temperatures must be in the recrystallization range to control fibering of TiN and TiC particles.

3. In this alloy, without "banding" or fibering it is possible to raise the Charpy V-notch toughness from 7 to 23 ft-lb at the same or higher strength levels.

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